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**60/201,923** **5 May 2000 (05.05.2000)** **US**
- (71) Applicant (*for all designated States except US*): **THE DOW CHEMICAL COMPANY [US/US]; 2030 Dow Center, Midland, MI 48674 (US).**
- (72) Inventors; and
- (75) Inventors/Applicants (*for US only*): **LADIKA, Mladen [US/US]; 4714 Blossom Circle, Midland, MI 48642 (US). FIBIGER, Richard, F. [US/US]; 3870 Johns Lane, Midland, MI 48642 (US). CHOU, Chai-Jing [US/US]; 1422 Bluestone Drive, Missouri, TX 77459 (US). BALAZS, Anna, C. [US/US]; 630 South Negley Avenue, Pittsburgh, PA 15232 (US).**
- (74) Agent: **ULMER, Duane, C.; The Dow Chemical Company, 2301 N. Brazosport Boulevard, Building B-1211, Freeport, TX 77541-3257 (US).**
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**WO 01/85831 A2**

(54) Title: **FUNCTIONALIZED POLYMER NANOCOMPOSITES**

(57) Abstract: A nanocomposite material containing several necessary components. The first component is from 98.9 to 70 parts by weight of a bulk polymer, the bulk polymer being a non-polar polymer, such as polyethylene or polypropylene. The second component is from 1 to 30 parts by weight of a cation exchanging multi-layered silicate material, such as acid or quaternary ammonium treated montmorillonite or sepiolite, dispersed in the bulk polymer, the cation exchanging multi-layered silicate material having a plurality of anionic sites, the multi-layered silicate material being exfoliated to one, two, three, four and more than four layer units, the number percent of the one, two, three and four layer units being greater than the number percent of the more than four layer units. The third component is from 0.1 to 10 parts by weight of an organic cation, such as polyethylene or polypropylene terminated by an amine group, the organic cation having a pendent polymer chain, the polymer of the pendent polymer chain being miscible with the bulk polymer, the average molecular weight of the pendent polymer chain being more than 3000. The nanocomposite material is made by blending the molten bulk polymer with the cation exchanging multi-layered silicate material and the organic cation.

## FUNCTIONALIZED POLYMER NANOCOMPOSITES

This application is under a United States Government  
5 contract with the Department of Commerce (NIST) - Advanced  
Technology Program Project #70NANB7H3028.

The instant invention relates to a composite material  
consisting of a delaminated or exfoliated cation  
10 exchanging multi-layered silicate material dispersed in a  
polymer. Such composite materials are known in the art as  
a "nanocomposite polymers" when at least one dimension of  
the exfoliated cation exchanging multi-layered silicate  
material is less than sixty nanometers. Nanocomposite  
15 polymers generally have enhanced mechanical property  
characteristics vs. conventionally filled polymers, for  
example, increased tensile or flex modulus together with  
increased impact toughness.

20 Typically, the thickness of a single layer of a  
delaminated cation exchanging multi-layered silicate  
material is in the range of one to two nanometers while  
the length and width of such layer can be in the range of,  
for example, one hundred to one thousand nanometers.  
25 Photomicrographs of nanocomposite polymers usually show a  
dispersion of multiple layer units of the cation  
exchanging multi-layered silicate material in the polymer,  
for example, two, three, four and more layer units  
dispersed in the polymer. It is generally desired to  
30 achieve a high degree of exfoliation of the cation  
exchanging multi-layered silicate material. Ideally the  
degree of such exfoliation is so extensive that only  
single layer units are present. However, some improvement  
may be seen even if the cation exchanging multi-layered

silicate material is only swelled with the bulk polymer, that is, "intercalated". If the cation exchanging multi-layered silicate material is not exfoliated or intercalated, then the mechanical property improvement of the polymer composite will usually be no better than if a conventional micron sized filler, for example, talc, is dispersed in the polymer.

Cation exchanging multi-layered silicate materials have been treated with organic onium ions to facilitate exfoliation when blended with polar polymers such as polyamide polymers, United States Patent 5,973,053, herein fully incorporated by reference. As discussed in the '053 patent, when such onium ion treated cation exchanging multi-layered silicate materials are blended with non-polar polymers (such as polyethylene or polypropylene) essentially no exfoliation occurs. However, as disclosed in the '053 patent, by incorporating more than ten percent of a polar substituted main guest molecule as a compatibalizer, it is possible to achieve an effective degree of exfoliation of the onium treated cation exchanging multi-layered silicate material into the non-polar polymer.

The use of the polar substituted main guest molecule of the '053 patent was an important advance in the art. However, more than ten percent of the relatively expensive polar substituted main guest molecule is required. It would be an advance in the art if a more effective compatabilizer were developed.

An important benefit of the instant invention is a solution to the above-mentioned problem. The instant invention is a nanocomposite material, comprising: a bulk

polymer, the bulk polymer being a non-polar polymer; a cation exchanging multi-layered silicate material dispersed in the bulk polymer, the cation exchanging multi-layered silicate material being exfoliated to one, 5 two, three, four and more than four layer units, the number percent of the one, two, three and four layer units being greater than the number percent of the more than four layer units, the cation exchanging multi-layered silicate content of the nanocomposite material being more 10 than one percent and less than thirty percent respectively by weight; and an organic cation, the organic cation having a pendent polymer chain, the polymer of the pendent polymer chain being miscible with the bulk polymer, the average molecular weight of the pendent polymer chain 15 being more than 3000, the organic cation content of nanocomposite material being more than one tenth of one percent and less than ten percent respectively by weight.

In another embodiment, the instant invention is a 20 process for making a nanocomposite material, comprising the step of blending from 98.9 to 70 parts by weight of a molten bulk polymer, 1 to 30 parts by weight of a cation exchanging multi-layered silicate material and 0.1 to 10 parts by weight of an organic cation to form the 25 nanocomposite material, the bulk polymer being a non-polar polymer, the organic cation having a pendent polymer chain, the polymer of the pendent polymer chain being miscible with the bulk polymer, the average molecular weight of the pendent polymer chain being more than 3000, 30 the multi-layered silicate material being exfoliated to one, two, three, four and more than four layer units, the number percent of the one, two, three and four layer units being greater than the number percent of the more than four layer units.

## BRIEF SUMMARY OF THE DRAWINGS

Fig. 1(a) shows a schematic drawing of a monovalent organic cation of the instant invention;

Fig. 1(b) shows a schematic drawing of a polyvalent  
5 organic cation of the instant invention;

Fig. 1(c) shows a schematic drawing of a polyvalent organic cation of the prior art; and

Fig. 2 shows a drawing of a photomicrograph of a nanocomposite material of the instant invention.

10

The instant invention is a nanocomposite material, comprising: (a) a bulk polymer, the bulk polymer being a non-polar polymer; (b) a cation exchanging multi-layered silicate material dispersed in the bulk polymer, the  
15 cation exchanging multi-layered silicate material being exfoliated to one, two, three, four and more than four layer units, the number percent of the one, two, three and four layer units being greater than the number percent of the more than four layer units, the cation exchanging  
20 multi-layered silicate material content of the nanocomposite material being more than one percent and less than thirty percent respectively by weight; and (c) an organic cation having a pendent polymer chain, the polymer of the pendent polymer chain being miscible with  
25 the bulk polymer, the average molecular weight of the pendent polymer chain being more than 3000, the organic cation content of the nanocomposite material being more than one tenth of one percent and less than ten percent respectively by weight. The term "miscible" in this  
30 regard means that the two polymers interpenetrate with each other. Ordinarily, the polymer of the pendent polymer chain is the same polymer type as the bulk polymer.

The bulk polymer can be a polyolefin such as polyethylene, polypropylene, polyvinylcyclohexane, hydrogenated polystyrene-butadiene-styrene, polypropylene-ethylene or polypropylene-butadiene. It should be understood that such polyolefins may be copolymers with C<sub>4</sub> to C<sub>10</sub> alpha-olefins, for example, polyethylene may be a copolymer of ethylene and 1-octene. The bulk polymer can also be polystyrene or ethylene-styrene interpolymer or any other non-polar polymer. The preferred polyolefin of the instant invention is polypropylene with or without the addition of maleated polypropylene. The polypropylene nanocomposite material of the instant invention can be blended with a thermoplastic elastomer to prepare the material known in the art as "thermoplastic olefin".

15

The term "cation exchanging multi-layered silicate material" is well known in the nanocomposite art and includes phyllosilicate clays and layered silicates. Illustrative of such materials are smectite clay minerals such as montmorillonite, nontronite, beidellite, hectorite, saponite, sauconite, and vermiculite clay minerals. This term also includes illite minerals such as ledikite and layered silicates such as magadiite and kenyaite. Preferred cation exchanging multi-layered silicate materials are phyllosilicates of the 2:1 type having a negative charge centers on the layers ranging from 0.25 to 1.5 charge centers per formula unit and a commensurate number of exchangeable cations in the interlayer spaces. Most preferred are smectite clay minerals such as montmorillonite, nontronite, beidellite, hectorite, saponite, sauconite, and the layered silicates magadiite, kenyaite, fluoromica and fluorohectorite.

30

The term "cation exchanging layered silicate material" also includes the layered fiber silicate materials such as attapulgite, boehmite, imogolite and sepiolite. Such materials exfoliate to produce multi-  
5 fiber units (herein multi-layer units) and most preferably they exfoliate to produce single fiber units (herein single layer units) dispersed in the polymer matrix.

The organic cation content of nanocomposite material  
10 is preferably more than one tenth of one percent and less than ten percent respectively by weight. The molecular weight of the organic cation is preferably in the range of from 5,000 to 300,000. The organic cation can be a polymer having a plurality of cationic sites or a single  
15 cation site such as an amine hydrochloride group, a quaternary ammonium group, a sulfonium group or a phosphonium group. However, the organic cation must have a "pendent polymer chain". The term "pendent polymer chain" means a polymer chain extending from a cation site  
20 that does not terminate with a cation site. Referring now to Fig. 1(a), therein is shown a polymer chain 10 having a molecular weight of 3,000 terminating in a nitrogen based cation group such as an amine hydrochloride or a quaternary ammonium group. The polymer chain 10 is a  
25 "pendent polymer chain". Referring now to Fig. 1(b), therein is shown a polymer chain 11 having a molecular weight of 15,000 terminating adjacent a nitrogen based cation group such as an amine hydrochloride or a quaternary ammonium group and then another polymer chain  
30 12 terminating adjacent another nitrogen based cation group. The polymer chain 11 is a "pendent polymer chain". Referring now to Fig. 1(c), therein is shown a polymer chain 13 terminating at each end with a nitrogen based cation group. The polymer chain 13 is not a "pendent

polymer chain" and is similar to the organic cation shown in Fig. 5 of United States Patent 5,973,053. The "pendent polymer chain" may be linear or branched.

5           The cation exchanging multi-layered silicate content of the nanocomposite material is preferably more than one percent and less than thirty percent respectively by weight. More preferably, the multi-layered silicate content of the nanocomposite material is more than two  
10 percent and less than twenty percent respectively by weight. Even more preferably, the multi-layered silicate content of the nanocomposite is more than three percent and less than ten percent respectively by weight.

15           Preferably, the cation exchanging multi-layered silicate has been treated with either an onium ion, a nonionic surfactant or hydrogen ion so that the onium ion and/or hydrogen ion is ionically bonded to more than one half of the anionic sites of the cation exchanging multi-  
20 layered silicate, the onium ion having a molecular weight of less than 3000. The specific onium ion used is not critical in the instant invention and can be any conventional onium ion as taught in, for example, United States Patent 5,973,053. More preferably, the cation  
25 exchanging multi-layered silicate has been treated with either an onium ion or hydrogen ion so that the onium ion and/or hydrogen ion is ionically bonded to ninety percent or more of the anionic sites of the cation exchanging multi-layered silicate. Such residual cation content of  
30 an acid and/or onium treated cation exchanging multi-layered silicate material can be determined by repeated washing with an acid solution followed by analysis of the wash solution for the amount of exchanged residual cation.



Referring now to Fig. 2, therein is shown a drawing reproduction of a photomicrograph of a nanocomposite polymer of the instant invention, showing a cross-sectional representative view of one, two, three, four, 5 five and twelve layer units of exfoliated cation exchanging multi-layered silicate material dispersed in the bulk polymer 14. The number of one, two, three and four layer units shown in Fig. 2 is fifteen. The number of more than four layer units shown in Fig. 2 is three 10 (two five layer units and one twelve layer unit). Therefore, the number percent of one, two, three and four layer units is greater than number percent of the more than four layer units (about 83 percent one, two, three and four layer units vs. about 17 percent more than four 15 layer units).

An organic cation of the type shown in Fig. 1(a) can be prepared using the hydroboration procedure outlined by Chung and Rhubright in J. Polym. Sci., Part A, Polym. 20 Chem., 1993, 31, 2759. An organic cation of the type shown in Fig. 1(b) can be prepared by reacting commercially available maleated polypropylene with N,N-dimethylethylenediamine in the presence of chlorobenzene at 130 degrees Celsius for 4 hours to produce an imine- 25 amine functionalized polypropylene that can then be converted to, for example, the amine hydrochloride form by reaction with hydrochloric acid.

The process of the instant invention comprises the 30 step of blending from 98.9 to 70 parts by weight of the molten bulk polymer, from 1 to 30 parts by weight of the cation exchanging multi-layered silicate material and from 0.1 to 10 parts by weight of the organic cation. It

should be understood that the process of the preceding sentence is practiced even when one or more components are pre-mixed. For example, when the cation exchanging multi-layered silicate material is pre-reacted with the organic cation to form an organic cation treated cation exchanging multi-layered silicate material which is then blended with the molten bulk polymer, or, for example, when the cation exchanging multi-layered silicate material has been treated with either an onium ion or a nonionic surfactant and then is pre-reacted with the organic cation to form an organic cation treated multi-layered silicate material which is then blended with the molten bulk polymer, or, for example, when the molten bulk polymer is blended with an amine terminated polymer and an acid treated cation exchanging multi-layered silicate material (the acid of the acid treated cation exchanging multi-layered silicate material reacting with the amine terminated polymer to produce the organic cation in situ, see Example 6 and 7 below).

20

## COMPARATIVE EXAMPLE 1

Ninety four grams of polypropylene (Montell grade PP-6524) and six grams of onium ion treated montmorillonite (available from the Southern Clay Company under the trade name CLAYTON HY) are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 60 rpm. X-ray diffraction and electron microscopic analysis of the resulting blend indicates little if any exfoliation of the onium treated montmorillonite. The resulting blend is then formed into test bars. The flex modulus of the resulting blend using ASTM method D-790 is 1,482,400 KPa (215,000 pounds per square inch).

30

## EXAMPLE 1

Ninety three grams of polypropylene (Montell grade PP-6524), six grams of onium ion treated montmorillonite (CLAYTON HY) and one gram of amine terminated 15,000 molecular weight polypropylene (the amine being in the hydrochloride form, see Fig. 1(a)) are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 60 rpm. X-ray diffraction and electron microscopic analysis of the resulting blend indicates substantial exfoliation of the onium treated montmorillonite into one, two, three, four and more than four layer units, the number percent of the one, two, three and four layer units being greater than the number percent of the more than four layer units. The resulting blend is then formed into test bars. The flex modulus of the resulting blend using ASTM method D-790 is 1,716,800 KPa (249,000 pounds per square inch).

## 20 COMPARATIVE EXAMPLE 2

Ninety four grams of polypropylene (Dow grade H705-04Z) and six grams of bis(octadecyl)dimethyl ammonium bromide treated magadiite are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 60 rpm. X-ray diffraction and electron microscopic analysis of the resulting blend indicates little if any exfoliation of the onium treated montmorillonite. The resulting blend is then formed into test bars. The flex modulus of the resulting blend using ASTM method D-790 is 1,261,800 KPa (183,000 pounds per square inch).

## EXAMPLE 2

Ninety three and nine tenths grams of polypropylene (Dow grade H705-04Z), six grams of the  
5 bis(octadecyl)dimethyl ammonium bromide treated magadiite and 0.1 gram of amine terminated 15,000 molecular weight polypropylene (the amine being in the hydrochloride form, see Fig. 1(a)) are blended at 180 degrees Celsius using a  
10 Haake Brand blender for 5 minutes at 60 rpm. X-ray diffraction and electron microscopic analysis of the resulting blend indicates significant exfoliation of the onium treated magadiite.

## EXAMPLE 3

15 Ninety three and eight tenths grams of polypropylene (Dow grade H705-04Z), six grams of the bis(octadecyl)dimethyl ammonium bromide treated onium ion treated magadiite and 0.2 gram of amine terminated 15,000 molecular weight polypropylene (the amine being in the  
20 hydrochloride form, see Fig. 1(a)) are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 60 rpm. X-ray diffraction and electron microscopic analysis of the resulting blend indicates significant exfoliation of the onium treated magadiite into one, two,  
25 three, four and more than four layer units, the number percent of the one, two, three and four layer units being greater than the number percent of the more than four layer units.

## 30 EXAMPLE 4

Ninety three and sixty five hundredths grams of polypropylene (Dow H705-04Z), six grams of the treated

magadiite and 0.35 gram of amine terminated 15,000 molecular weight polypropylene (the amine being in the hydrochloride form, see Fig. 1(a)) are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 60 rpm. X-ray diffraction and electron microscopic analysis of the resulting blend indicates significant exfoliation of the onium treated magadiite into one, two, three, four and more than four layer units, the number percent of the one, two, three and four layer units being greater than the number percent of the more than four layer units. The resulting blend is then formed into test bars. The flex modulus of the resulting blend using ASTM method D-790 is 1,420,400 KPa (206,000 pounds per square inch).

15

#### EXAMPLE 5

Ninety three and one half grams of polypropylene (Dow grade H705-04Z), six grams of the bis(octadecyl)dimethyl ammonium bromide treated magadiite and 0.5 gram of amine terminated 15,000 molecular weight polypropylene (the amine being in the hydrochloride form, see Fig. 1(a)) are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 60 rpm. X-ray diffraction and electron microscopic analysis of the resulting blend indicates significant exfoliation of the onium treated magadiite into one, two, three, four and more than four layer units, the number percent of the one, two, three and four layer units being greater than the number percent of the more than four layer units.

30

#### COMPARATIVE EXAMPLE 3

Ninety seven grams of polypropylene (Dow grade H705-04Z) and three grams of acid treated montmorillonite

(Fluka grade K 20) are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 60 rpm. Electron microscopic analysis of the resulting blend indicates little if any exfoliation of the acid treated magadiite. The resulting blend is then formed into test bars. The flex modulus of the resulting blend using the ASTM method D 790 is 1,406,600 KPa (204,000 pounds per square inch). The impact toughness of the resulting blend using ASTM method is 1.25 joules (11.1 inch pounds).

10

## EXAMPLE 6

Ninety six grams of polypropylene (Dow grade H705-04Z), three grams of acid treated montmorillonite (Fluka grade K 20) and one gram of amine terminated 15,000 molecular weight polypropylene are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 60 rpm. Electron microscopic analysis of the resulting blend indicates significant exfoliation of the onium treated magadiite into one, two, three, four and more than four layer units, the number percent of the one, two, three and four layer units being greater than the number percent of the more than four layer units. The resulting blend is then formed into test bars. The flex modulus of the resulting blend using ASTM method D-790 is 1,461,700 KPa (212,000 pounds per square inch). The impact toughness of the resulting blend using ASTM method is 1.76 joules (15.6 inch pounds).

30

## EXAMPLE 7

Ninety five grams of polypropylene (Dow grade H705-04Z), three grams of acid treated montmorillonite (Fluka grade K 20) and two grams of amine terminated 15,000 molecular weight polypropylene are blended at 180 degrees

Celsius using a Haake Brand blender for 5 minutes at 60 rpm. Electron microscopic analysis of the resulting blend indicates significant exfoliation of the onium treated magadiite into one, two, three, four and more than four  
5 layer units, the number percent of the one, two, three and four layer units being greater than the number percent of the more than four layer units. The resulting blend is then formed into test bars. The flex modulus of the resulting blend using ASTM method D-790 is 1,537,600 KPa  
10 (223,000 pounds per square inch). The impact toughness of the resulting blend using ASTM method is 2.06 joules (18.2 inch pounds).

#### COMPARATIVE EXAMPLE 4

15 Thirty and six tenths grams of polypropylene (Amoco grade 9934) and five and four tenths grams of onium ion treated sepiolite (bis(hydrogenated tallow) dimethyl ammonium chloride treated) are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 150  
20 rpm. An electron microscopic analysis of the resulting blend indicates little exfoliation of the onium treated sepiolite. The resulting blend is then formed into test bars. The tensile modulus (using a test method ASTM D 638) of the resulting blend is 2,9167,000 KPa (423,000  
25 pounds per square inch).

#### EXAMPLE 8

Twenty seven grams of polypropylene (Amoco grade 9934), five and four tenths grams of onium ion treated  
30 sepiolite (bis(hydrogenated tallow) dimethyl ammonium chloride treated), one and eight tenths grams of octadecanamide, and one and eight tenths grams of amine terminated 15,000 molecular weight polypropylene (the amine being in the hydrochloride form, see Fig. 1 (a)) are

blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 150 rpm. An electron microscopic analysis of the resulting blend indicates substantial exfoliation of the onium treated sepiolite into one, two, three, four, and more than four fiber units, the number percent of one, two, three, and four fiber units being greater than the number percent of the more than four fiber units. The resulting blend is then formed into test bars. The tensile modulus (using a test method ASTM D 638) of the resulting blend is 3,399,000 Kpa (493,000 pounds per square inch).

#### COMPARATIVE EXAMPLE 5

Thirty and six tenths grams of polypropylene (Amoco grade 9934) and five and four tenths grams of onium ion treated sepiolite (dimethyl dioctadecyl ammonium bromide treated) are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 150 rpm. An electron microscopic analysis of the resulting blend indicates little if any exfoliation of the onium treated sepiolite. The resulting blend is then formed into test bars. The tensile modulus (using a test method ASTM D 638) of the resulting blend is 3,241,000 KPa (470,000 pounds per square inch).

#### EXAMPLE 9

Twenty seven grams of polypropylene (Amoco grade 9934), five and four tenths grams of onium ion treated sepiolite (dimethyl dioctadecyl ammonium bromide treated), one and eight tenths grams of maleated polypropylene (Eastman, Epolene Brand, grade G-3003), and one and eight tenths grams of amine terminated 15,000 molecular weight polypropylene (the amine being in the hydrochloride form, see Fig. 1 (a)) are blended at 185 degrees Celsius using a



Haake Brand blender for 5 minutes at 150 rpm. An electron microscopic analysis of the resulting blend indicates substantial exfoliation of the onium treated sepiolite into one, two, three, four, and more than four fiber units, the number percent of one, two, three, and four fiber units being greater than the number percent of the more than four fiber units. The resulting blend is then formed into test bars. The tensile modulus (using a test method ASTM D 638) of the resulting blend is 3,599,00 KPa (522,000 pounds per square inch).

#### COMPARATIVE EXAMPLE 6

Thirty two and six tenths grams of polypropylene (Amoco grade 9934) and seven and four tenths grams of onium ion treated fluormica (bis(hydrogenated tallow) dimethyl ammonium chloride treated) are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 100 rpm. The resulting blend is then formed into test bars. The tensile modulus (using a test method ASTM D 638) of the resulting blend is 3,055,000 KPa (443,000 pounds per square inch).

#### EXAMPLE 10

Twenty eight and six tenths grams of polypropylene (Amoco grade 9934), seven and four tenths grams of onium ion treated fluormica (bis(hydrogenated tallow) dimethyl ammonium chloride treated), two grams of maleated polypropylene (Eastman, Epolene Brand, grade G-3003), and two grams of amine terminated 15,000 molecular weight polypropylene (the amine being in the hydrochloride form, see Fig. 1 (a)) are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 100 rpm. The resulting blend is then formed into test bars. The tensile modulus (using a test method ASTM D 638) of the

resulting blend is 3,689,000 KPa (535,000 pounds per square inch).

#### COMPARATIVE EXAMPLE 7

5           Thirty two and six tenths grams of polypropylene (Amoco grade 9934) and seven and four tenths grams of onium ion treated fluormica (bis(hydrogenated tallow) dimethyl ammonium chloride treated) are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes  
10 at 100 rpm. The resulting blend is then formed into test bars. The flex modulus (using a test method ASTM D 790) of the resulting blend is 2,475,000 KPa (359,000 pounds per square inch).

#### 15 EXAMPLE 11

          Twenty eight and six tenths grams of polypropylene (Amoco grade 9934), seven and four tenths grams of onium ion treated fluormica (bis(hydrogenated tallow) dimethyl ammonium chloride treated), two grams of maleated  
20 polypropylene (Eastman, Epolene Brand, grade G-3003), and two grams of amine terminated 15,000 molecular weight polypropylene (the amine being in the hydrochloride form, see Fig. 1 (a)) are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 100 rpm. The  
25 resulting blend is then formed into test bars. The flex modulus (using a test method ASTM D 790) of the resulting blend is 2,896,000 KPa (420,000 pounds per square inch).

#### COMPARATIVE EXAMPLE 8

30           Thirty two and six tenths grams of polypropylene (Dow grade DC 112) and seven and four tenths grams of onium ion treated fluormica (bis(hydrogenated tallow) dimethyl ammonium chloride treated) are blended at 180 degrees Celsius using a Haake Brand blender for 5 minutes at 100

rpm. The resulting blend is then formed into test bars. The flex modulus (using a test method ASTM D 790) of the resulting blend is 1,538,000 KPa (223,000 pounds per square inch).

5

## EXAMPLE 12

Twenty eight and six tenths grams of polypropylene (Dow grade DC 112), seven and four tenths grams of onium ion treated fluormica (bis(hydrogenated tallow) dimethyl ammonium chloride treated), two grams of maleated  
10 polypropylene (Eastman, Epolene Brand, grade G-3003), and two grams of amine terminated 15,000 molecular weight polypropylene (the amine being in the hydrochloride form, see Fig. 1 (a)) are blended at 180 degrees Celsius using a  
15 Haake Brand blender for 5 minutes at 100 rpm. The resulting blend is then formed into test bars. The flex modulus (using a test method ASTM D 790) of the resulting blend is 2,241,000 (325,000 pounds per square inch).

WHAT IS CLAIMED IS:

1. A nanocomposite material, comprising:
  - (a) a bulk polymer, the bulk polymer being a non-polar polymer;
  - 5 (b) a cation exchanging multi-layered silicate material dispersed in the bulk polymer, the cation exchanging multi-layered silicate material being exfoliated to one, two, three, four and more than four layer units,  
10 the number percent of the one, two, three and four layer units being greater than the number percent of the more than four layer units, the cation exchanging multi-layered silicate content of the nanocomposite  
15 material being more than one percent and less than thirty percent respectively by weight; and
  - (c) an organic cation, the organic cation having a pendent polymer chain, the polymer  
20 of the pendent polymer chain being miscible with the bulk polymer, the average molecular weight of the pendent polymer chain being more than 3000, the organic cation content of nanocomposite material  
25 being more than one tenth of one percent and less than ten percent respectively by weight.
2. The nanocomposite material of Claim 1,  
30 wherein the bulk polymer is selected from the group consisting of polyethylene, polypropylene and polyvinylcyclohexane, polystyrene and an ethylene-styrene interpolymers.

3. The nanocomposite material of Claim 2,  
blended with a thermoplastic elastomer to  
make a thermoplastic olefin.
- 5 4. The nanocomposite material of Claim 2,  
wherein the cation exchanging multi-layered  
silicate material is selected from the  
group consisting of montmorillonite,  
nontronite, beidellite, volkonskoite,  
10 hectorite, saponite, sauconite, magadiite,  
octasilicate, fluoromica, fluorohectorite,  
kenyaite, attapulgite, boehmite, imogolite  
and sepiolite.
- 15 5. The nanocomposite material of Claim 2,  
wherein the organic cation content of the  
nanocomposite material is more than one  
tenth of one percent and less than ten  
percent respectively by weight and wherein  
the cation exchanging multi-layered  
silicate content of the nanocomposite  
20 material is more than two percent and less  
than twenty percent respectively by weight.
- 25 6. The nanocomposite material of Claim 4,  
wherein the organic cation content of the  
nanocomposite material is more than two  
tenths of one percent and less than six  
percent respectively by weight and wherein  
the cation exchanging multi-layered  
silicate content of the nanocomposite  
material is more than three percent and  
30 less than ten percent respectively by  
weight.
7. A process for making a nanocomposite  
material, comprising the step of blending

5 from 98.9 to 70 parts by weight of a molten  
bulk polymer, 1 to 30 parts by weight of a  
cation exchanging multi-layered silicate  
material and 0.1 to 10 parts by weight of  
an organic cation to form the nanocomposite  
material, the bulk polymer being a non-  
polar polymer, the organic cation having a  
pendent polymer chain, the polymer of the  
pendent polymer chain being miscible with  
10 the bulk polymer, the average molecular  
weight of the pendent polymer chain being  
more than 3000, the multi-layered silicate  
material being exfoliated to one, two,  
three, four and more than four layer units,  
15 the number percent of the one, two, three  
and four layer units being greater than the  
number percent of the more than four layer  
units.

- 20 8. The process of Claim 7, wherein the bulk  
polymer is selected from the group  
consisting of polyethylene, polypropylene  
and polyvinylcyclohexane, polystyrene and  
an ethylene-styrene interpolymer.
- 25 9. The process of Claim 8, wherein the  
nanocomposite material is blended with a  
thermoplastic elastomer to make a  
thermoplastic olefin.
- 30 10. The process of Claim 8, wherein the cation  
exchanging multi-layered silicate material  
is selected from the group consisting of  
montmorillonite, nontronite, beidellite,  
volkonskoite, hectorite, saponite,  
sauconite, magadiite, octasilicate,

fluoromica, fluorohectorite, kenyaite, attapulgite, boehmite, imogolite and sepiolite.

- 5                   11. The nanocomposite material of Claim 8, wherein the organic cation content of the nanocomposite material is more than one tenth of one percent and less than ten percent respectively by weight and wherein the cation exchanging multi-layered
- 10                   silicate content of the nanocomposite material is more than two percent and less than twenty percent respectively by weight.
- 15                   12. The process of Claim 10, wherein the organic cation content of the nanocomposite material is more than two tenths of one percent and less than six percent respectively by weight and wherein the cation exchanging multi-layered silicate
- 20                   content of the nanocompsosite material is more than three percent and less than ten percent respectively by weight.

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FIG. 1A

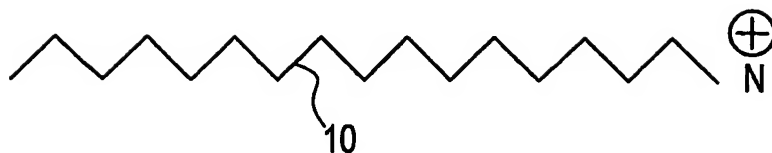


FIG. 1B

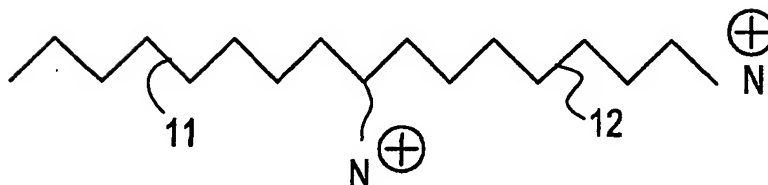
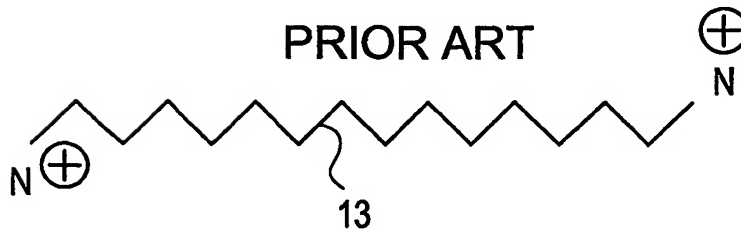


FIG. 1C

PRIOR ART





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FIG. 2

